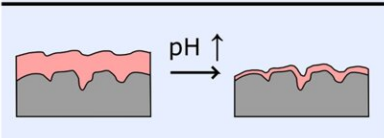
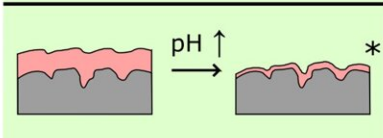
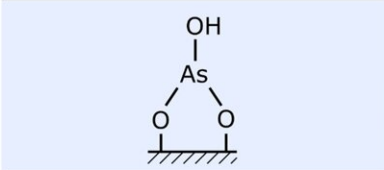
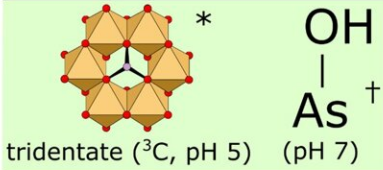
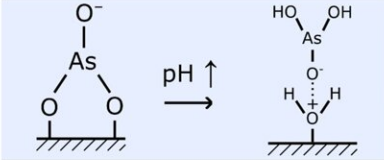
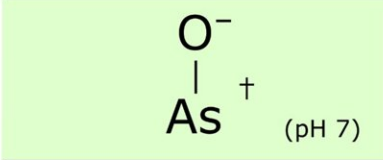
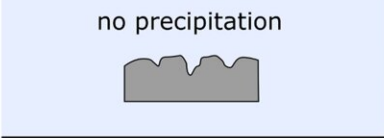
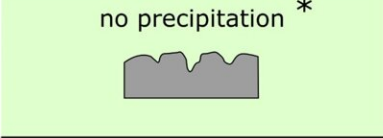
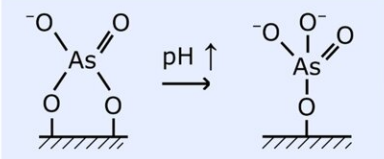
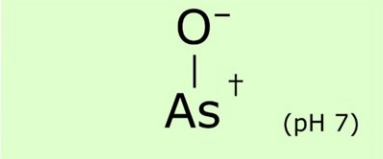
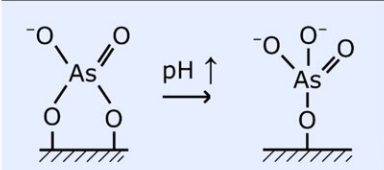
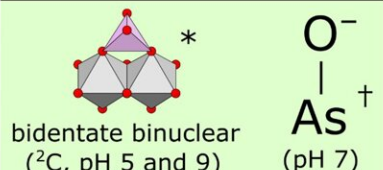


# A single-step water treatment for arsenic decontamination

November 25 2022

	Adsorption mechanism	Surface Complexation Model	Experiment (EXAFS*, ζ+, FTIR†)
As(III)	precipitation		
	Fe <sub>2</sub> O <sub>3</sub>		 tridentate ( <sup>3</sup> C, pH 5) (pH 7)
	TiO <sub>2</sub>		 O <sup>-</sup> As <sup>+</sup> (pH 7)
As(V)	precipitation	no precipitation 	no precipitation* 
	Fe <sub>2</sub> O <sub>3</sub>		 O <sup>-</sup> As <sup>+</sup> (pH 7)
	TiO <sub>2</sub>		 bidentate binuclear ( <sup>2</sup> C, pH 5 and 9) (pH 7)

The authors combined spectroscopic data obtained at Diamond Lightsource

(EXAFS) with FTIR and zeta potential measurements at Imperial College London to develop a picture of the dominant surface complexes formed when arsenic is adsorbed by TiO<sub>2</sub>/Fe<sub>2</sub>O<sub>3</sub> composite nanomaterials. Arrows indicate where the pH is increased from 5 to 9. Asterixes (\*) and daggers † indicate evidence gained by EXAFS and FTIR/zeta potential analysis respectively. Credit: Imperial College London and Diamond Light Source

A team of researchers from Imperial College London led by Prof. Dominik Weiss has been working with Diamond Light Source, the UK's national synchrotron on a new material (*TiO<sub>2</sub>/Fe<sub>2</sub>O<sub>3</sub> nanomaterial*) combining photocatalytic oxidation with adsorption, which allows a one-step treatment of contaminated water.

Over 50 million people in South Asia are exposed to groundwater contaminated with carcinogenic [arsenic](#). The team's results were recently published in *Results in Surfaces and Interfaces* outlining how the high versatility of Diamond's B18 beamline allowed them to perform X-ray Absorption Spectroscopy on their samples and compare the results with their [predictive models](#).

The researchers already have a patent for this material for arsenic decontamination and they hope that the [new material](#) might be incorporated into a filtration column for everyday use in affected households, as this cheap and efficient technology could improve the quality of water for millions of people. .

First author of the publication, Dr. Jay Bullen explains, "These experiments allowed us to directly investigate the structure of the surface complexes formed when arsenic binds to our TiO<sub>2</sub>/Fe<sub>2</sub>O<sub>3</sub> nanomaterial. We were able to conclude that the surface complexes we had chosen for our predictive surface complexation model, using pure titania and pure

iron oxide reference materials, were appropriate."

"However, we also saw some tridentate As-Fe<sub>2</sub>O<sub>3</sub> bonding in the EXAFS data that wasn't included within our earlier model. We were also able to confirm surface precipitation of As(III) at low pH, which our model had predicted."

Arsenic is a famously toxic element and long-term exposure to even trace amounts can lead to debilitating and potentially fatal diseases including skin cancer, lung cancer, keratosis, and neurological disorders. 100-200 million people globally are believed to be exposed to arsenic through contaminated groundwater drinking supplies. Water multi-step treatments to remove arsenic already exist, using filtration (adsorption), but this process requires pre-treatment (oxidation) to be efficient .

Dr. Jay Bullen says, "We wanted to better understand the nature of arsenic adsorbed onto this composite TiO<sub>2</sub>/Fe<sub>2</sub>O<sub>3</sub> nanomaterial; to understand whether arsenic binds in the same way as onto pure TiO<sub>2</sub> and pure Fe<sub>2</sub>O<sub>3</sub> minerals."

"X-ray Absorption Spectroscopy (XAS) is the perfect way to do this, with EXAFS revealing information about the local environment of arsenic atoms, e.g. how many [covalent bonds](#) arsenic forms with the TiO<sub>2</sub>/Fe<sub>2</sub>O<sub>3</sub> surface. The beamtime grant we received allowed us to realize these experiments at Diamond Lightsource."

## **Spectroscopic observations to confirm a predictive model**

Arsenic can be found in several forms in water including arsenite and arsenate. Arsenite (i.e. As(III)) ions are difficult to remove from contaminated water using conventional treatments such as adsorption or coagulation-flocculation due to its neutral charge (H<sub>3</sub>AsO<sub>3</sub>). In contrast,

arsenate (i.e. As(V)) ions ( $\text{HAsO}_4^{2-}$  and  $\text{H}_2\text{AsO}_4^-$ ) are both (i) more easily adsorbed and (ii) less toxic than  $\text{H}_3\text{AsO}_3$ .

Consequently, the treatment of As(III)-contaminated water benefits from the oxidation of As(III) to As(V) prior to its removal via adsorption. Oxidation of As(III) can be achieved using heterogeneous photocatalysts, such as  $\text{TiO}_2$ , and ultraviolet (UV) radiation. However, other materials such as iron oxides ( $\text{Fe}_2\text{O}_3$ ) are more efficient for adsorption. Therefore multiple-step treatment plants are currently used but their design and maintenance is challenging.

This difficulty may be overcome by incorporating photocatalytic and adsorption capabilities into a single material. Previous studies by the same group showed that composite materials combining the excellent photocatalytic capabilities of  $\text{TiO}_2$  with the high As(V) adsorption capacities of iron oxides ( $\text{Fe}_2\text{O}_3$ ) were good candidates for efficient decontamination.

The authors subsequently developed a surface complexation model (SCM) to predict changes in the amount of arsenic adsorbed and its speciation as a function of experimental variables such as pH.

However, the authors wanted to verify that the structures of the adsorbed arsenic surface complexes chosen for the model were realistic, so they used spectroscopic techniques. X-ray absorption spectroscopy played a key role thanks to its element selectivity, its capability to discriminate As oxidation state (XANES) and to identify the structure of the As complexes adsorbed on the  $\text{TiO}_2/\text{Fe}_2\text{O}_3$  surface (EXAFS).

The team used B18 to realize their experiments. At the beamline, they were able to record EXAFS spectra of solid samples to determine how many covalent bonds arsenic forms with the surface of the materials, and XANES spectra of aqueous suspensions to measure photooxidation

kinetics.

The authors combined spectroscopic data obtained at Diamond Lightsource (EXAFS) with FTIR and zeta potential measurements at Imperial College London to develop a picture of the dominant surface complexes formed when arsenic is adsorbed by  $\text{TiO}_2/\text{Fe}_2\text{O}_3$  composite nanomaterials.

This was used to evaluate and verify the speciation of adsorbed arsenic predicted by the authors' previous Surface Complexation Model (SCM), a theoretical model that is able to predict changes in adsorption as a function of environmental conditions. This model had been developed using surface complexes chosen based on data for pure titania and pure iron oxides, with experimental evidence for arsenic speciation on composite  $\text{TiO}_2/\text{Fe}_2\text{O}_3$  not being available prior to the Diamond Lightsource study.

**More information:** Jay C. Bullen et al, Spectroscopic (XAS, FTIR) investigations into arsenic adsorption onto  $\text{TiO}_2/\text{Fe}_2\text{O}_3$  composites: Evaluation of the surface complexes, speciation and precipitation predicted by modelling, *Results in Surfaces and Interfaces* (2022). [DOI: 10.1016/j.rsurfi.2022.100084](https://doi.org/10.1016/j.rsurfi.2022.100084)

Provided by Diamond Light Source

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